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Characteristics of Fe-, Ni- and Co-based powder coatings fabricated by laser metal deposition without preheating the base material.

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Abstract

The objective of this work was to select the best material from Fe-, Ni- and Co-based alloy powder for coating, by Laser Metal Deposition (LMD) the filets of a hardened 42CrMoS4 extrusion screw without preheating process. Even though most of the articles recommended preheating the base material as a condition for a crack free coating, the time wasted in the process decrease the productivity and distortions can be also generated in the part. In this work, a comparison of the main characteristics of the coatings done on preheated and non-preheated base material has been made.
The relationships between the relevant LMD parameters (feed rate, laser power, and powder feeding rate) and the main geometrical characteristics of a single clad (height, width, dilution, deposition rate, efficiency, etc.) were examined. In addition, different characteristics of overlapped clads in a preheated, non-preheated and a hardened base material have been also analyzed.
All the study was made in the Ibarmia ZVH 45/1600 Add+Process hybrid machine with a high power Yb-Fiber laser (3 kW) and discrete coaxial LMD head. Coatings with thickness from 1.2 to 0.76 were created without cracks and other defects except in the case of Ni-based coating. The microstructural features of these coatings were studied using optical and scanning electron microscopy. The mechanical properties were determined using microhardness measurements and a pin on disk tribometer.

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Keywords: Laser metal deposition; coatings; non-preheat; geometrical characteristics; Co-Based; Fe-Based; Ni-Based

1. Introduction

The threads of rubber extrusion screws are subjected to high wear during their life-cycle due to high temperatures and high friction suffered during the extrusion process. Thus, hard coatings are commonly used to increase substantially the life-cycle of such screws. In addition, the coating can be replaced and brought back to tolerance after the coating has suffered excessive erosion, reducing the need for the production of new extrusion screws.

Despite there are several techniques for hard coatings production, such as PVD, PTA, HVOF [1], Laser Metal Deposition (LMD) process by powder injection has been demonstrated to be effective and flexible technology for this application.

The LMD process applies a laser heat source to deposit a thin layer of a desired metal on the surface of a selected substrate. The resulting mixture changes the surface properties and the substrate becomes a composite material with improved properties.

The commonly used materials for this kind of applications are Ni-based, Co-based or martensitic Fe-based alloys in powder form. These materials are used because of its properties like high hardness, excellent wear resistant and/or thermal resistance. In this regard, the works of De Hosson *et al.* [2-3], Ocelík *et al.* [4-5] and Bartkowski *et al.* [6-7] clarify LMD parameters, coating microstructure, hardness and common defects like pores or cracks on Ni- and Co-based coatings.

Preheat and slow cold down processes are common solutions to produce crack free coatings in low weldability

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materials [4, 5, 8, 9]. The LMD process is not an exception, although this process produces high quality coatings with a minimal heat input into the work piece. This pre-heating process leads to a significant decrease in the productivity of the coating process, not only because of the pre-heating time, but also because the processing part needs to be moved to an oven. In addition, this part handling can result also in a difficult and costly task when it comes to large size pieces.

On the other hand, the release of new hybrid machines which combines the LMD technology with machining processes, open new manufacturing options which allows machining the screws and adding the hard coatings in the same set-up, without the need of moving the part from one machine to the other in order to perform the different processes (machining of part and addition of coating). Because of a great variety of powdered materials with excellent properties can be used for LMD of wear resistant coatings, to find a material that can be deposited without preheating is the key to implement a continuous hybrid process without moving the processing part, thus, increasing the productivity and simplifying the process to the use of a single machine.

The final objective of this study is to manufacture high-resistant coatings without preheating the base material, in order to be able to manufacture or repair rubber extrusion screws in a hybrid machine. For that, this study presents an experimental study using three different powder materials (Ni-based, Co-based and Fe-based) and three different states of the base material (non-treated, pre-heated and hardened). The parameters of the three best clads have been used to build coatings for each material. In addition to the geometrical and defects requirements, the mechanical properties have been tested in the built coats.

2. Experimental Procedure

2.1. Materials

The material used as a substrate material was a turned 42CrMoS4 steel alloy bar of 50 mm in diameter and 160 mm in length. The base material was in three different states: non-treated, pre-heated and hardened

Hardening started with a heat process in an oven at 850ºC for 2.5h, quenched in oil, tempered at 250ºC for 2.5h and cooled down slowly at room temperature. The hardness was measured before and after the hardening process, obtaining a hardness of 33 and 52 HRC respectively.

The powders used to produce the coatings were: 1) Eutroloy 16606A (Fe-based); 2) Eutroloy 16006N (Co-based, equivalent to Stellite 6 powder); and 3) Colmonoy 56 P2 (Ni-based).

All powders were characterized in terms of size distribution determined by laser diffraction, morphology determined by SEM, fluidity according to ISO 4490 standard and chemical analysis. Low fluidity, low sphericity and small powder grain size have consequences of producing irregular powder mass flow rate. In this case, the powders presented the required characteristics (Table 1).

Table 1- Properties of powder materials

	EUTROLOY 16606A	*EUTROLOY 16006N*	*COLMONOY 56 P2*
Shape	*spherical*	*spherical*	*spherical*
Size (μm) d_{50}	*113*	*90*	*102*
Fluidity (s/50g)	*11.9*	*10.9*	*12.9*
Density (g/cm³)	*7.9*	*8.44*	*8.18*

2.2. Hybrid (LMD+machining) machine

All tests were performed in an IBARMIA ZVH45/1600 Add+process hybrid machine (Figure 1). This multiprocess machine combines the additive manufacturing based in LMD technology with 5 axis milling and turning capacity. This machine is equipped with a Precitec YC52LMD discrete coaxial head with 4-stream nozzle and with 2 mm powder focus diameter. The system also uses a Sulzer Metco TWIN-10-C Powder Feeder and an Yb-Fiber Rofin FL030 Laser generator of 3kW and with a continuous wavelength of 1.07 μm.

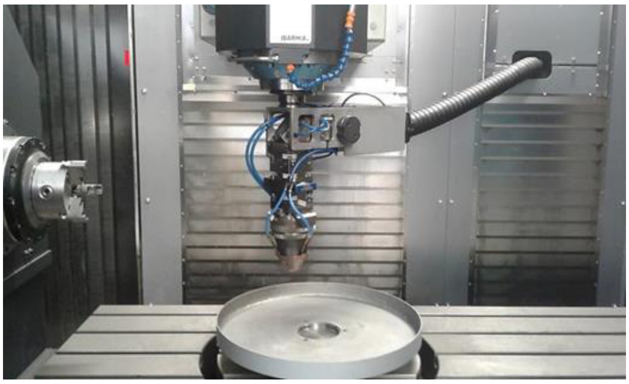


Figure 1. Ibarmia ZVH45/1600 add+process.

2.3. Experimental Testing

The experimental testing consisted on first, performing single tests at *different* conditions of laser power (P), feed rate (F) and powder mass flow rate ($\dot{m}_p$) for selecting three optimum process combinations for each coating material. Then, the selected combinations of parameters were validated by performing overlapped tests.

All tests were performed with the laser beam focused at 12.5 mm above the substrate surface giving a circular beam size of 2.25 mm in diameter.

The substrate material was cleaned and degreased before the additive process with acetone and centered with a roundness of ±0,01mm deviation. The bar was rotated in the horizontal turning spindle. Because the powder feeder rate is regulated by a rotating wheel, the parameter that defines it is the percentage of the maximum rotation. The rotation percentage defines different powder mass flow rate depending of the fluidity and density of the powder material. The relation between the powder feeder rotating wheel speed percentage and the powder mass flow rate was measured by collecting and weighting the powder delivered in three minutes. The results are shown in Table 2.

Table 2-Powder feeder calibration

Rotation percentage	Powder mass flow rate, ṁp (g/s)		
	Eutroloy 16606A	Eutroloy 16006N	Colmonoy 56 P2
50%	0.37	0.35	0.29
75%	0.52	0.50	0.44
100%	0.67	0.66	0.58

2.3.1. Single clads

In order to select optimum process parameters for each coating material, first, single clads were carried out at all parameters combinations stated in Table 3, resulting in a total of 18 single clads for each coating material and each base material condition (non-treated, pre-heated and hardened).

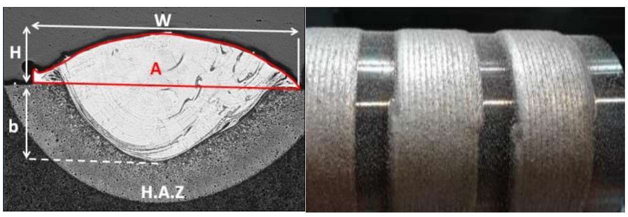
Table 3-Process main parameters

Laser power **P (W)**	Powder feeder rotation percentage	Feed rate **F (mm/min)**
1500	50%	750
2000	75%	1000
2500		1250

All clads have been observed in the microscope for observing the microstructural integrity of the deposition using optical and scanning electron microscopy. Samples have been chemically etched by Vilella etching. Some samples corresponding to Eutroloy 16006N clads needed a harder chemical treatment, and a Chromic acid has been employed. In addition to the microstructural integrity, each single clad has been measured geometrically in terms of height (H), width (W), penetration (b) and area (A) by employing image analyzer software (Clemex Captiva®), as shown in Figure 2-a. The dilution (d), the Material Deposition Rate (MDR) and the powder efficiency (ε) were calculated using the following equations:

$$MDR = A \cdot \rho \cdot F \quad (1) \qquad \varepsilon = \frac{MDR}{\dot{m}p} \quad (2)$$

$$d\ (\%) = \left(\frac{b}{b+H}\right) \cdot 100 \quad (3)$$



(a) (b)

Figure 2. (a) Single clad Macrophotography; (b) overlapped clad.

2.3.2. Overlapped clads

The analysis of single clads served to select three optimum processing conditions for each coating material. In all cases with the exception in the case of Colmonoy 56 P2 for the pre-heated and non-treated case, the optimum parameters consisted on powder feed rate correspondent to a 50% of the powder feeder rotating speed, 2.5 kW of laser power and three different feed rates (750-1000-1250 mm/min). In the latter, the selected laser power was 2kW. These conditions were used to fabricate overlapped clads by rotating the steel bar meanwhile the laser track moved along the traversal axis (Figure 2-b). To obtain a uniform thickness, all the coatings were created with 50% overlapping of the single clad width. All the conditions were proved in three kinds of substrate; non-heat treated, preheated and hardened 42CrMoS4 cylinder. In the overlapped clads, in addition to the characteristics measured and calculated in the single clads (H, W, b, MDR, ε and d), the productivity rate (Pr) was also calculated as the surface area coated per minute, which can be calculated using the equation 4.

$$Pr = w \cdot (1 - overlap) \cdot F \quad (4)$$

In order to validate the efficiency calculated using the equation 2, the cylinder was weighted after and before of the cladding process for measuring the deposited clad weight and compare it to the theoretical added powder.

In addition, mechanical properties of the overlapped clads were also determined by using microhardness tester and a pin on disk tribotester at 100ºC temperature for the wear test. A standard pin on disk test with an AISI 52100 pin (60-66 HRC) and 5N load was done. For the pin on disk test, all clads were deposited on hardened substrate and all the samples were ground before be tested.

2.4. Preheating Process setup

The laser beam was used to preheat the cylinder up to a temperature of 257ºC (measured using a thermocouple). The pre-heating temperature was calculated employing the Séférian method [10].

The chuck was thermally isolated with thermal ceramic inserts to prevent damages in the spindle. To avoid the surface melting, the laser was focused at 100mm to increase the size of the spot and decrease the energy density. The desired temperature was reached in 30 minutes but the cooling process lasted more than 1.5h.

3. Results and Discussion

3.1. Single Clads analysis

All single clads were cracks free, including tests carried out without pre-heating the base material. However, some pores have been observed in some cases due to excess of powder. The pores quantity decreased when increasing the laser power and when reducing the powder mass flow rate. No pores were found in the case of Eutroloy 16006N when using 2500W of laser power and 50% of rotation speed of the powder feeder. In the case of Colmonoy 56 P2 the pores quantity is reduced also increasing the laser power and reducing the powder mass flow rate and the feed rate. However, the generated pores were bigger, which is in agreement with the results obtained by Aubry *et al.* [8].

3.1.1. Relationships between clad characteristics and process parameters

For the same laser spot size and substrate, same relationships between the single clads characteristics and the main parameters (P and F) were found for all materials analyzed. The dependence with such parameters and the type of trend for each characteristic is summarized in Table 4. Some examples of these relationships are shown in Figure 3. The results showed that for each material and the same powder mass flow rate, the variation of height in clads with the same feed rate and different power is relatively low and increases at higher speeds. The width has a strong dependence of the laser power; it behaves contrary to the height. Efficiency and deposition rate behaves like the width. . In addition, the efficiency seems to reach a maximum value at a certain value of $P^{3/2}/F$. Single clad area (area excepting the dilution zone) behaves like the height but with linear trend and the sum of penetration and height is related to the ratio between the laser power and the feed rate, thus, to the energy density (equation 5). Where E is the energy density in J/mm^2 and D is the laser spot diameter in mm.

Table 4-Dependence of the single clads parameters

Clad Characteristic	*Dependence parameter*	*Type of trend*
Width	$\frac{\sqrt{P^3}}{F}$	*Logarithmic*
Efficiency and deposition rate	$\frac{\sqrt{P^3}}{F}$	*Polynomial*
Height	$\frac{\sqrt{P}}{F}$	*Logarithmic*
Area	$\frac{\sqrt{P}}{F}$	*Linear*
Height+penetration	$\frac{P}{F}$	*Logarithmic*

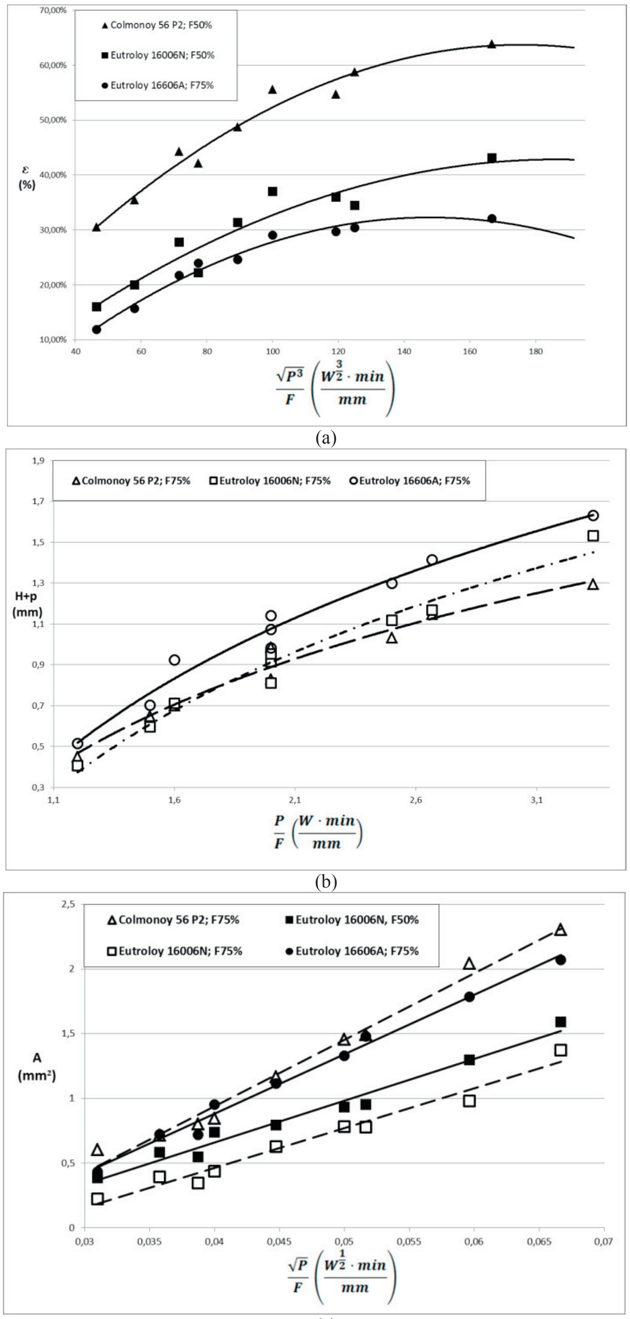
$$E = \frac{P}{F \cdot D} \qquad (5)$$

3.2. Overlapped clads analysis

Clads overlapped at 50% presented a continuous thickness (Figure 5). In the case of Eutroloy coating materials (both Co- and Fe-based) high quality clads have been obtained. Colmonoy 56 P2 presented defects in all the cases including some cracks at 1000 mm/min in hardened and non-preheated cases, showed less pores in preheated and hardened cases.

Actually, for a laser spot of 2.25 mm diameter and a powder focus diameter of 2mm, it was found that using energy per powder mass (obtained dividing the laser power by the powder mass flow rate) higher than 6800 J/g no pores were found in Eutroloy material coating overlapped at 50% (both Fe- and Co-based), when using 2500W of laser power and 50% of rotation speed of the powder feeder.

Table 5 summarizes the results obtained for all materials optimum process parameters on hardened substrate.



(a) (b) (c)

Figure 3. Examples of relationships between single clad characteristics and process parameters for the: (a) efficiency; (b) height+penetration; and (c) area.

Table 5- Results of overlapped clads tests

HARDENED SUBSTRATE							
Nº	F (mm/min)	H (mm)	b (mm)	MDR (kg/h)	ε (%)	d (%)	Pr (mm^2/min)
Eutroloy 16606A	750	1.15	0.34	0.59	45.01	22.68	1091.25
Eutroloy 16606A	1000	0.86	0.38	0.61	45.96	30.66	1480.00
Eutroloy 16606A	1250	0.76	0.41	0.57	43.49	34.85	1581.25
Eutroloy 16606N	750	1.20	0.06	0.74	57.95	5.03	1211.25
Eutroloy 16606N	1000	1.03	0.05	0.72	56.50	4.24	1380.00
Eutroloy 16606N	1250	0.85	0.09	0.72	56.64	9.62	1.662.50
Colmonoy 56 P2	750	1.16	0.15	0.72	68.34	11.19	1273.13
Colmonoy 56 P2	1000	0.94	0.26	0.75	71.16	21.97	1635.00
Colmonoy 56 P2	1250	0.78	0.31	0.75	70.86	28.33	1950.00

3.2.1. Comparison between clads obtained with non-treated, pre-heated and hardened base material

The efficiency and the coating height in preheat and hardened cases are very similar and higher than in non-treated case.

In preheated case, the Heat Affected Zone (HAZ) was deeper. All the coatings presented higher dilution and less height in the first and second overlapped clad, being continuous after the third clad.

Eutroloy 16606A presented defects free structure. Eutroloy 16006N (Co-based) presented no defects and low geometrical dilution in all cases. A scanning electron microscope (SEM) analysis was needed to check the welding zone in the case of Eutroloy 16006N showing a good quality welded zone of the Co-based material and to test the chemical dilution in all the materials. Colmonoy 56 P2 has fewer pores in preheated substrate.

A martensitic structure was observed in the HAZ of non-treated substrate. High dilution of Fe in Colmonoy 56 P2 coating was detected creating NiFe, Ni2Fe and Ni3Fe intermetallic alloys that decrease the hardness of the clad in pre-heated case. In the case of Eutroloy 16006N a high dilution was detected in the first clad but not in the others. That explains the different color of the first Eutroloy 16006N clad in the macrophotography (figure 5).

Regarding the efficiency, very similar values have been obtained in three conditions tested for each powder material and each substrate state (non-treated, preheated and hardened). Thus, the efficiency and consequently, the MDR, are independent to the feed rate. In addition, since the clad width also varies too little with the feed rate, there exists an inversely proportional relationship between the coating height and feed rate, in order to maintain a constant MDR. Therefore, there exists a linear relationship between the coating height and productivity rate (figure 4): for the same processing conditions of laser power and powder mass flow rate, the height decreases linearly with the productivity rate. This relationship between the height and the productivity rate summarizes clearly the coating results that can be obtained for each processing condition and each material. As can be seen in figure 4, in the present study the hardened substrate provided the best results in terms of productivity rate and coating height.

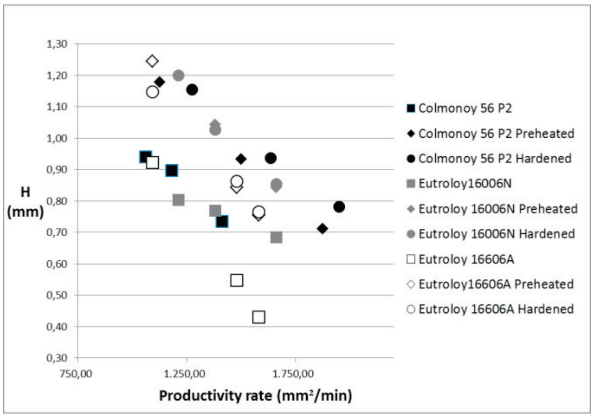


Figure 4. Relation between height and productivity rate for all the coatings in the three type of substrate.

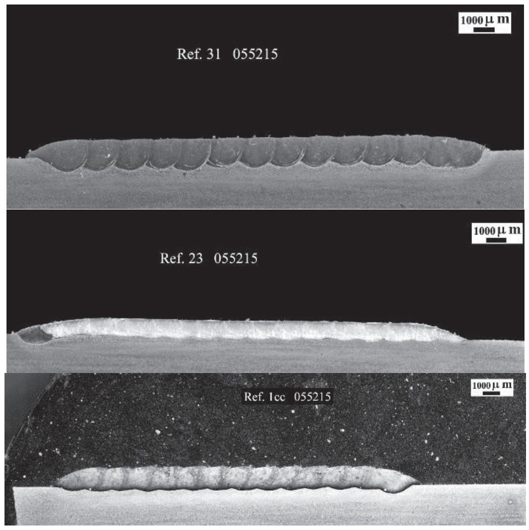


Figure 5. 50% overlapped coatings thickness. Ref. 31: Eutroloy 16606A, Ref. 23: Eutroloy 16006N, Ref. 1cc: Colmonoy 56 P2.

3.2.2. Wear and hardness properties of the overlapped coatings

The HAZ in non-heat treated case had high hardness due to the hardening of the substrate and, in the hardened substrate, the HAZ showed less hardness due to tempering process.

The average values of clad hardness are shown in table 6 and pin on disk wear testing results in table 7. The substrate (42CrMoS4) had a hardness of 52 HRC after hardening and 33 HRC in the other cases. Eutroloy 16606A presented a similar coating hardness in all the cases but a bit higher in pre-heated and hardened cases. Eutroloy 16006N (Co-based) presented lower hardness in the non-treated case. Eutroloy 16006N and Colmonoy 56 P2 have higher hardness in hardened substrate. Colmonoy 56 P2 has a low hardness in preheated substrate due to a high chemical dilution.

Regarding the results obtained in the pin on disk tests. Eutroloy 16606A coated sample and the 42CrMoS4 hardened control substrate show a good wear resistance. The friction coefficient value is due to the high wear of the pin that increases the contact surface. The wear value in the case of Colmonoy 56 P2 and Eutroloy 16006N is far away from the other results. Colmonoy 56 P2 showed worse wear possibly due to internal pores.

Table 6-Hardness mean value.

Hardness HRC									
Substrate	*Non-treated*			*Hardened*			*Pre-heated*		
F (mm/s)	*750*	*1000*	*1250*	*750*	*1000*	*1250*	*750*	*1000*	*1250*
Colmonoy 56P2	*56*	*51*	*53*	*57*	*55*	*55*	*50*	*31*	*31*
Eutroloy 16606A	*60*	*56*	*-*	*62*	*59*	*63*	*63*	*61*	*62*
Eutroloy 16006N	*45*	*42*	*42*	*54*	*54*	*52*	*52*	*53*	*50*

Table 7-Pin on disk wear testing results.

Wear rate			
Sample	*Wear track* $(mm^3/(N{\cdot}m))$	*Wear pin* $(mm^3/(N{\cdot}m))$	*Friction coefficient*
42CrMoS4	*-*	*5.42E-05*	*0.84*
Colmonoy 56 P2	*4.08E-04*	*1.01E-05*	*0.60*
Eutroloy 16606A	*-*	*8.15E-05*	*0.91*
Eutroloy 16006N	*3.38E-04*	*1.70E-06*	*0.54*

4. Conclusions and future work

This study demonstrated that coating by laser metal deposition a hardened 42CrMoS4 extrusion screw without preheating process and zero defects is possible except with Colmonoy 56 P2. Defect free coatings in Eutroloy 16606A (Fe-based) and 16006N (Co-based) have been obtained at 2500W of laser power and 50% of powder feeder rate.

Regarding the single clad analysis of the present work, strong dependence between clad characteristics and the laser power and the feed rate have been observed. This analysis should be extended to other materials and substrates. In addition, the relation between single clad characteristics and multilayer and overlapped clads should be also investigated, carrying out an exhaustive study of the parameters variability and process repeatability. On the other hand, an inflection point of the powder efficiency where it reaches its maximum value has been found in single clad tests. A deeper study of this trend should be carried out in order to find a working range for high efficiency coating process.

Regarding the analysis done for the overlapped clads, following conclusions have been obtained:

- The best coatings were obtained with a hardened 42CrMoS4 substrate, in terms of hardness, powder efficiency, productivity rate and coating height.
- A homogenous height was generated with coatings overlapped at 50% of the single clad width.
- In the coatings, the first and second clad showed more dilution and less height.
- The efficiency increases in the hardened and preheated substrate.
- Colmonoy 56 P2 hardness is lower in the preheated case due to higher dilution.
- The HAZ in the hardened case showed less hardness due to tempering process of the substrate caused by the laser beam when depositing the coating.

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